GREEN SYNTHESIS, CHARACTERISATION AND APPLICATIONS OF TRANSITION METAL OXIDE NANOCOMPOSITES USING MICROWAVE ASSISTED METHOD

SYNOPSIS

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1. Introduction

1.1 Nanotechnology

The concept of nanotechnology is defined by Richard Feynman, a Nobel Prize winner, in 1959. He states that "there's Plenty of Room at the Bottom", "where he discussed the possibilities of manipulating individual atoms" (Sao et al., 2010). In 1974, Professor Norio Taniguchi has used the term nanotechnology and defined the term as "processing, separation, consolidation and deformation" of bulk material through atoms or molecules. However in 1980s, K Eric Drexler has formally coined the term, "Nanotechnology". At present it is collaborating with all fields of science with its multiple applications for the welfare of mankind.

1.2 Nanoparticles

Nanoparticles (NPs) (1–100 nm in size) have unique properties due to their small size, large surface area and quantum effects, distinguishing them from bulk materials. These characteristics make them valuable in a wide range of applications, including medicine, energy, agriculture and consumer products.

NPs are extensively studied for their distinct chemical and physical characteristics, including electronic, mechanical, magnetic, dielectric, thermal, optical and biological properties. These features open up a wide array of applications in emerging sectors of nanoscience and nanotechnology, particularly in environmental, biomedical and biotechnological domains (Singh et al., 2019).

Since the size, shape and surface morphology NPs of are critical factors that significantly influence their functionality and potential applications. Due to their high surface energy, NPs are highly reactive and tend to aggregate if their surfaces are not stabilized or capped (Khataee et al., 2014).

In the current era of semiconductor advancement, metal oxide NPs has garnered significant interest from researchers and technologists due to their exceptional electrical and optical characteristics. Besides, their extremely small size significantly influences their optical and thermal properties also (Gunalan et al., 2012). Therefore, its smaller size and high surface-to-volume ratio make it possible to utilize it in other applications for enhancing the strengths and capabilities of materials.

1.3 Metal Oxide Nanoparticles

Metal oxide NPs are composed of metal and oxygen atoms, exhibit unique size-dependent properties and high surface area, enhancing their reactivity. These features make them valuable in applications like catalysis, where they serve as efficient catalysts and make them valuable across diverse applications. For example TiO_2, ZnO, CuO, NiO and Fe_2O_3 are used in respective fields like cosmetics, sensors and environmental remediation. Despite their wide-ranging applications, metal oxide NPs pose concerns related to toxicity and environmental impact. This has spurred extensive research to ensure safe and sustainable use.

Metal oxide NPs has derived from metal precursors. Precise control over their facets, size and shape is vital for advanced material applications. While early research focused on carbon based nanomaterials, transition metal ions like Au, Ag, Zn, Cu, Ni, Co and Ti have shown promise in catalytic and antioxidant applications. However due to high cost of some metals like gold and titanium, more accessible alternatives are being explored. Hitherto some important metal oxide NPs are explored.

Zinc as a transition metal with atomic number 30 is the 24th most abundant element in Earth's crust. Zinc oxide primarily exists in three crystal forms as Wurtzite, zinc blende and rock salt with hexagonal Wurtzite structure is being most stable. In this form zinc atoms are tetrahedrally coordinated with four oxygen atoms. Each structural variation functions as a semiconductor, characterized by a direct wide band gap of approximately 3.3 eV. The notable band gap of ZnO NPs ranging from 3.3 to 3.4 eV, positions them for applications in various optoelectronic devices such as light-emitting diodes, photodetectors and p-n junctions. ZnO NPs exhibit a substantial binding energy of 60 meV which enhances luminescence efficiency even at room temperature. ZnO NPs exhibit visible photoluminescence (PL) emission around 2.5 eV (~495 nm), attributed to intrinsic defects. This optical property makes them ideal for use in field emission and vacuum fluorescent displays. Such applications have been highlighted in recent studies (Narender et al., 2022; Ribbing and Roos, 1997). ZnO NPs are recognized for their affordability and diverse properties encompassing sensing, catalysis, dye degradation and antimicrobial activity (Rani et al., 2020; Rathod et al., 2017).). The reduction of zinc nitrate hexahydrate, in the presence of cetyl trimethyl ammonium bromide (CTAB) as capping agent and further varied the morphology of particles i.e. from rod to spherical shape has been obtained with certain amount of CTAB. The photocatalytic results of

synthesized NPs show that the material with the higher band gap (E_g) values are more efficient (Suwanboon et al., 2011). These advantageous characteristics coupled with their economic and safe attributes, make it highly suitable for applications in sensors, transparent conductors, organic transformations and surface acoustic wave devices. Notably, ZnO NPs play a pivotal role in cosmetics, effectively absorbing a wide spectrum of radiation and providing protection against harmful ultraviolet rays. Furthermore, ZnO NPs find significant utility in photocatalytic and biomedical applications including biomedical imaging, drug delivery, gene delivery and bio sensing (Aboulouard et al., 2020; Bhatia and Verma, 2017; Danish et al., 2021). ZnO NPs have strong adsorption capacity and catalytic efficiency make them valuable in both environmental and industrial settings.

Further as CuO exhibit both monoclinic and cubic structures which enhance their functional performance. With a band gap typically falling within the semiconductor range, approximately between 1.2 to 1.7 eV, CuO NPs exhibit notable characteristics. Their antimicrobial activity has led to applications in antibacterial coatings, medical devices and water purification systems. The catalytic behavior of CuO NPs contributes to enhanced reaction rates and selectivity, making them pivotal in catalysis processes. Leveraging their electrical and optical properties, particularly their band gap, CuO NPs find their utility in electronics, sensors and optoelectronics. In biomedical fields, these NPs play a role in wound healing, drug delivery and imaging applications (Yadav et al., 2022).

Meanwhile Cobalt oxide (Co_3O_4) NPs have diverse industrial applications, including use in Li-ion battery anodes, catalysts, gas sensors and magnetic materials (Jalali et al., 2023). Their resistance to corrosion and oxidation makes cobalt NPs valuable in everyday technologies. Common forms include cobalt oxide (II) (CoO) with a rock-salt structure and cobalt oxide (III) (Co_2O_3) exhibiting distinct properties. These NPs have garnered attention across multiple fields, particularly in catalysis, electronics and energy storage devices. CoO NPs, with their unique electronic properties and high surface area are explored for catalytic applications, while Co_3O_4 NPs present potential in both catalysis and electronic systems. Further their magnetic properties make them suitable for applications in data storage and sensors. These properties of cobalt oxide NPs varying with size and shape, influencing their performance in different applications. This

tunability drives innovation in fields like energy storage, sensing and catalysis. Ongoing research explores their potential across diverse technological domains.

Besides, Nickel oxide (NiO) NPs have diverse applications due to their unique properties. They act as effective antibacterial agents due to their surface reactivity and oxidative stress generation. With their high surface area and redox activity, NiO NPs serve as efficient catalysts in chemical reactions (Avinash et.al., 2021). Their electrical conductivity and optical properties make them valuable in sensors for detecting gases and pollutants. NiO NPs wide band gap and stability are beneficial for solar cells and batteries. In biomedical fields, they exhibit antioxidant and cytotoxic effects, showing potential in cancer treatment. However, their safety for medical use requires further research.

Recently, Manganese oxide MnO NPs are studied in the series of transition metals, which is used for their electrochemical**,** catalytic**,** and biomedical properties. As manganese is among the most abundant element of the earth and found twelfth most common element on the planet and the third most abundant transition element after iron and titanium. However, less attention has been paid to manganese as a high-performance metal in various applications such as medicine, biomedicine, biosensors, water treatment and purification, electronics, electrochemistry, photoelectronics, catalysis etc. Manganese oxides has wealthy structures such as MnO, Mn_5O_8, Mn_2O_3, MnO_2 and Mn_3O_4, which can be used in a variety of fields (Hoseinpour and Ghaemi, 2018). So that MnO NPs potentially hold great promise for sustainable nanotechnology.

Consequently recent trends indicated that the Metal oxides and their composites are among the important material groups which are attracting the attention of researchers due to their wide application areas. Therefore transition metals near to Zn have been studied for their promising future in nanocomposites.

1.4 Nanocomposites

Nanocomposites (NCs) are an advanced class of materials created by embedding NPs into a matrix to enhance performance. These NPs, usually smaller than 100 nanometers, interact synergistically with the host material. This results in improved mechanical, thermal, electrical, or chemical properties. NCs are classified into different types such as polymer NCs, metal matrix NCs, metal-metal oxide NCs and ceramic matrix NCs (Ghoreishi et al., 2013). Their superior mechanical, thermal and electrical properties

make them valuable in advanced material engineering. The modification of transition metal oxide-based NPs introduces a new array of properties, including increased surface area, enhanced surface activities, improved porosity, reduced electron-hole recombination and heightened conductivities (Dey et al., 2015).

While transition metal oxides refer to compounds composed of transition metals and oxygen. Transition metals are elements found in the d-block of the periodic table, characterized by the filling of inner d-orbitals as electrons are added. These metals often exhibit multiple oxidation states, leading to the formation of diverse oxides with distinct properties. Transition metal oxides play crucial roles in various fields due to their electronic, magnetic, catalytic and structural properties (Yadav et al., 2022). Therefore to study transition metal oxide nanocomposites NCs are particularly notable for their diverse properties and wide-ranging potential.

Zinc oxide nanocomposites (ZnO) NCs combine ZnO NPs with other materials to enhance their properties and expand their applications. ZnO is known for its wide band gap, high excitation binding energy and strong antimicrobial properties. These features make ZnO NCs suitable for use in sensors, catalysis, electronics, medical devices, optoelectronics and coatings. Their unique characteristics, including high transparency in the visible range, strong UV absorption, semiconducting behavior and high thermal stability, make them highly effective for diverse application (Dey et al., 2015).

Copper oxide nanocomposites (CuO) NCs, including cupric oxide (CuO) and cuprous oxide (Cu_2O) are valued for their distinct chemical and physical properties. CuO, with a monoclinic structure and a band gap of 1.2-1.9 eV, is known for its high thermal stability and excellent electrical properties. On the other hand, Cu_2O, with a cubic structure and a band gap of 2.0-2.2 eV, is noted for its good photoconductivity. These properties make CuO and Cu_2O NCs ideal for use in catalysis, sensor technology, energy storage, lithium-ion batteries and supercapacitors. Recent research trends focus on enhancing their properties through composite materials, exploring environmental applications like water treatment and pollutant degradation and investigating their potential in biomedical fields, including drug delivery and medical diagnostics (Rane et al., 2018).

Cobalt oxides, encompassing cobalt (II) oxide (CoO) and cobalt oxide (III) (Co_3O_4) NCs are advanced materials that incorporate cobalt oxide NPs with other substances to enhance their properties for a variety of applications. Co_3O_4 is a mixed-valence

compound of cobalt with a spinel structure while CoO is a simple cubic oxide, both of which exhibit unique physical and chemical properties such as high thermal stability, catalytic activity and excellent electrochemical properties. They are also utilized in magnetic applications, benefiting from their ferromagnetic properties. Therefore due to vast properties of cobalt oxide NCs, including high surface area, robust catalytic activity, sensing, environmental remediation, and tunable electronic properties, make them suitable for advanced applications (Mondal et al., 2020).

Further, Nickel Oxide (NiO) NCs are advanced materials formed by integrating NiO NPs with other functional materials to enhance their physical, chemical, and electronic properties. As a p-type semiconductor with a wide band gap (3.6–4.0 eV), NiO exhibits excellent thermal stability, high electrochemical activity, and strong catalytic behavior. These properties make it a highly versatile component in the development of nanocomposites. Thus due unique properties of NiO NCs including high thermal stability, excellent chemical resistance, significant surface area, and tunable electrical conductivity, making them suitable for high-performance applications (Lin and Chen, 2014).

Similarly, Manganese oxides can be used in molecular sieves, photovoltaic cells, light emitting diodes, drug delivery ion sieves, and a variety of other applications, including imaging contrast agents, magnetic hard disks, food sciences and water purification. There are different ways to synthesize Mn based zinc nanocomposites by using sole gel process, microwave radiation etc. but there is a dire need for scientific research on the eco-friendly synthesis of Mn doped ZnO NCs and their application in the biological arena (Hasan et al., 2024).

In present decade Scientists are dedicated to enhancing the effectiveness, efficiency and durability of practical applications for NCs by skillfully combining appropriate constituents in the synthesis process (Kannan et al., 2020). In recent research, NCs based on transition metal oxides have garnered significant attention as versatile and promising materials across various scientific fields, owing to their broad spectrum of practical applications. Numerous studies in the literature have highlighted the utility of transition metal oxide-based NCs in diverse applications. For instance, Carbon nanotube-ZnO NCs have been identified as an electrode material for supercapacitors, exhibiting a calculated specific capacitance of 323.9 (Dey et al., 2015). Additionally, synthesized CuO/ZnO NCs have been explored for their antibacterial properties (Shafey., 2020), while ZnO/CuO NCs have demonstrated humidity sensor capabilities (Jeevanandam et al.,

2022). These studies underscore the broad range of applications and potential advancements in the field of transition metal oxide based NCs in recent years. In photocatalytic applications, they facilitate efficient light absorption and charge separation while biomedical applications, such as drug delivery, antibacterial and imaging, also leverage the unique properties of metal oxide NCs. As researchers refine synthesis methods, controlling size, shape and composition, the landscape of these NCs continues to expand, promising advancements in materials and science and technology (Patra and Baek, 2014) in which synthesizing methodology is most important.

2. Literature Review

The green method for synthesis of the ZnO NPs have been reported by using different parts of number of plants. The green synthesis of the ZnO NPs probably found to be initiated by the use of aloe-vera plant by (Sangeetha et al., 2011) respectively. Ravindra et al., 2011 , have used altered amounts of Latex of Maddar (Calotropis procera) for the synthesis of ZnO NPs with 5-40 nm size and spherical/granular shape. The synthesis and characterization of metal oxide-based NCs, particularly those incorporating Zinc, have accumulated significant attention in recent years due to their unique properties and potential applications across various fields. By examining their synthesis methods, structural characteristics and performance attributes highlight the latest advancements and applications across various fields, including catalysis, energy storage and environmental remediation. The subsequent paragraphs provide a concise literature review to comprehend the synthesis, characterization and applications of metal oxide NCs.

Gurgur et al. (2020) have synthesized ZnO, ZnO-Ag and ZnO-Cu nanomaterials by using dye extracted from *Bridelia ferruginea* leaf extract with enhanced properties while minimizing adverse environmental effects. The nanomaterial's crystallinity was assessed, resulting in 19.02 nm for ZnO and 18.90 nm for Cu-ZnO. The synthesized nanomaterials exhibited different morphologies. The optical band gap of 3.24 eV for the ZnO nanoparticles. Which can be narrowed to 3.18 and 3.13 eV by silver and copper dopant respectively. These results showed the nanoparticles as a potential agent for photovoltaics and other optical applications

Medina-Ramirez et al. (2019) highlighted the significance of Cu@ZnO NCs as a notable material category with versatile applications encompassing catalysts, photocatalysts,

optoelectronic devices and antimicrobial agents because of its high redox activity. This study specifically investigated the antibacterial effectiveness of Cu@ZnO NCs, subjecting them to evaluation against E. coli and S. aureus bacteria.

Takele et al. (2023) have synthesized CuO, ZnO and CuO/ZnO NCs by using *Zingiber officinale* rhizome extract. XRD analysis revealed the crystalline nature of all synthesized particles, with particle sizes of 4.35, 14.54, 18.41 and 20.50 nm for CuO NPs, ZnO NPs, 10 % CuO/ZnO and 20 % CuO/ZnO NCs respectively. The highest inhibition zones against Staphylococcus aureus and Escherichia coli were achieved by 20 % CuO/ZnO NCs, with diameters of 20 + 0.7 and 16 + 0.5 mm, respectively, at a concentration of 50 mg/mL.

Zhou et al. (2021) synthesized a core-sheath nanostructured Cu/Cu20-ZnO-Fe304 material to tackle challenges associated with chlorine-resistant bacteria (CRB) and disinfection byproducts (DBPs) in tap water post-chlorination. The NCs exhibited exceptional antibacterial efficacy against CRB while maintaining low cytotoxicity levels, attributed to its unique nanowire structure and the generation of reactive oxygen species. Moreover, it displayed impressive photocatalytic degradation of trichloroacetic acid (TCAA) under simulated sunlight, with surface reactions dominating the process. Interestingly, the NCs showed reduced cytotoxicity compared to individual components, with higher cell growth rates observed after incubation.

Fang et al. (2018) synthesized urchin-like CuO/ZnO NCs via a biomimetic hydrothermal process with subsequent calcination, employing glutamine (Gln) as a growth regulator. The resulting hierarchical structure exhibited closely packed and interconnected NPs, with sizes ranging from 20 to 50 nm. Gln played a pivotal role in determining the phase composition and morphology of the NCs. Comparative analyses revealed that the urchin-like CuO/ZnO NCs, facilitated by Gln, displayed exceptional efficiency in degrading methylene blue dye under UV irradiation compared to ZnO/CuO nanosheets without GIn and pure ZnO with GIn. The morphological and compositional characteristics of the NCs directly influenced their photocatalytic properties, with the urchin-like structure demonstrating the highest efficiency.

Chang et al. (2016) synthesized a novel Cu/ZnO core/shell nanostructure, comprised of Cu NPs with an average diameter of 44.4 + 4.3 nm, enveloped by a finely tuned layer of ZnO measuring 4.8 + 0.5 nm in thickness. The remarkable result of their work was the

efficient photocatalytic performance of these Cu/ZnO core/shell nanomaterials. Notably, these nanomaterials displayed the ability to degrade the pollutant methylene blue, a capability that was notably absent in pure copper nanoparticles. These findings highlighted the immense potential of Cu/ZnO core/shell heterostructures as a promising and effective solution for catalyzing the degradation of pollutants through photocatalysis.

Igbal et al. (2022) synthesized a NCs of Cu-doped ZnO, incorporating polyacrylic acid (PAA) microgel to tackle the degradation of a colored pollutant using sunlight. The process involved doping ZnO nanostructures with varying amounts of copper (ranging from I to 20 %) through the co-precipitation technique. This synthesized material was used to enhance its photocatalytic degradation under sunlight.

Modwi et al. (2017) have synthesized Cu/ZnO NCs via a sol-gel method. Characterization revealed a wurtzite phase with crystallite sizes ranging from 15.86 to 24.25 nm and a mesoporous structure with pore diameters from 24.63 to 52.88 nm. Adsorption and degradation studies demonstrated enhanced performance of Cu/ZnO compared to pure ZnO, with 23.55 % and 14.49 % malachite green (MG) adsorption on Cu/ZnO and ZnO, respectively. The Langmuir model fits well for Cu/ZnO adsorption, with a 1539 mg/g capacity. Cu/ZnO exhibited a degradation rate constant of 10 x 10~ min™, about 8 times higher than ZnO (1.28 x 10 min™").

Bekru et al. (2022) synthesized NCs of CuO-ZnO by using an aqueous eVerbascum sinaiticum Benth. Authors have reported the degradation efficiency of CuOZnO NCs against MB dye, with a rate constant of 0.017 min' compared to 0.0027 min™! for ZnO NPs. Similarly, a ratio constant of 5.925 min g for 4-NP reductions was achieved.

Meena et al. (2021) have synthesized NCs of ZnO/CuO/Ag:O via a chemical method. Authors reported the photocatalytic efficacy of ZnO/CuO/Ag:O was evaluated against the degradation of crystal violet and rhodamine B organic dyes under solar light.exposure. Results revealed about 99.05 and 97.38 % degradation efficiencies for CV and RhB dyes within 105 minutes.

Habibi and Rezvani (2015) investigated the photocatalytic degradation of C.I. Reactive Red 195 using a copper cobaltite NCs coated on glass, fabricated through the Doctor Blade method. Characterization revealed well-defined NPs with a size of 31 nm, showcasing a favorable nanoscale crystalline morphology. The NCs exhibited high efficiency, degrading over 85 % of the dye within 45 minutes of irradiation, with a rate

constant of 4.10 x 10 min™' determined through kinetic modeling. Notably, the developed photocatalyst overcomes challenges associated with separation and recycling, enhancing its practical feasibility for large-scale water treatment processes. These findings highlight the potential of copper cobaltite NCs as sustainable and effective solutions for water purification applications.

Elavarasan et al. (2022) synthesized Co_3O_4/ZnO-Cu NCs using a simple hydrothermal method and evaluated their photocatalytic efficacy for Rhodamine Blue (Rh B) dye degradation under simulated light. The Co_3O_4/ZnO-Cu NCs exhibited exceptional performance, achieving a remarkable degradation efficiency of 95.33 % within 100 minutes, surpassing both Co_3O_4 and ZnO-Cu counterparts. The kinetic rate constant of Co_3O_4/ZnO-Cu NCs was found to be 6.76 times higher than bare Co_3O_4, indicating efficient catalytic activity. Moreover, the NCs demonstrated excellent recyclability maintaining 90.7 % dye degradation efficiency even after four consecutive cycles.

Ethiraj et al. (2020) investigated the photocatalytic degradation of phenolic compounds using Cu-doped nickel oxide (Cu-NiO) nanocatalysts. The study revealed the superior efficiency of Cu-NiO compared to pure NiO in phenol degradation, with Cu doping influencing crystallinity, energy bandgap values, and particle size. Morphological analyses confirmed smaller particle sizes in Cu-NiO, indicating the impact of copper doping. Successful application in real industrial effluent, particularly in leather industrial wastewater, demonstrated an impressive removal efficiency of 85.7 % within 150 minutes.

Lin and Chen (2014) elaborated that NiO NCs have a broad range of applications due to their unique properties. In energy storage, they are used in supercapacitors and batteries, where their high electrochemical activity enhances energy density and cycle stability. In catalysis, NiO NCs serve as efficient catalysts for reactions like water splitting and organic transformations, benefiting from their high surface area and active sites. Additionally, they are utilized in gas sensors, where their sensitivity to gases such as hydrogen and methane is leveraged for environmental monitoring and safety. NiO NCs also find applications in optoelectronics, including light-emitting diodes (LEDs) and solar cells, due to their optical and electronic properties.

Hassan et al. (2024) investigated Mn-doped ZnO nanoparticles are prepared by using the reducing and capping components of the plant extract *W. coagulans*. The basic purpose

of this research is to fabricate, characterize and biologically evaluate the Mn doped ZnO nanoparticles. This paper confirms via the nanoparticle's crystalline stability and its smaller size (11–14 nm) through XRD. thereafter EDX of different ratios 2:1, 4:1, 1:1, 1:2 of Mn doped ZnO nanoparticles gives the outcomes of 27.0%, 10.8%, 55.1%, 10.1%, 27.7%, 23.3%, and 7.4%, 13.0% respectively. The antibacterial activity was performed, showing more activity against the gram-positive bacterial strain S. aureus and less against the gramnegative bacteria E. coli. Overall, it was shown that biologically fabricated nanoparticles are more stable, crystalline, smaller, and active. Additionally, they showed greater antibacterial and photocatalytic activity against the bacteria and dye, respectively.

3. Research Gap Identification

Metal oxide (NCs) possess tunable properties and enhanced functionalities, making them valuable in diverse fields such as nanotechnology, materials science, energy, healthcare and environmental science. Despite significant progress, wide research gaps are remaining, particularly in the development of innovative synthesis techniques to broaden the scope of biomedical and photocatalytic applications. These gaps include the need for precise control over nanoparticle properties, improved stability and durability, increased multifunctionality and the reduction of toxicity and environmental impact. Although there are various methods for the synthesis of different metal oxide NCs but the microwave-assisted green method using medicinal plant extracts with different ratios of d-block (3d-series) metal precursors would likely to be the most important eco friendly synthesis and has not been explored at large. Therefore it will be innovative to explore microwave-assisted synthesis of NCs in photocatalytic, antioxidant and antimicrobial applications.

4. Objectives

(i) To synthesize different metal oxide NCs of different compositions by using the microwave-assisted green method.

(ii) To characterize the synthesized NCs by employing various spectroscopic techniques.

(iii) To explore the photocatalytic, antioxidant and antifungal/antibacterial activities of the synthesized NCs.

5. Proposed Methodology

5.1 Materials

5.1.1 Chemicals

The chemicals such as zinc nitrate hexahydrate, copper(II) nitrate trihydrate, cobalt(II) acetate tetrahydrate, nickel(II) nitrate hexahydrate, manganese(II) nitrate hexahydrate, sodium hydroxide pellet, methyl orange, methylene blue, malachite green, sulfuric acid, ethanol, methanol, hydrogen peroxide, nitric acid, potassium dichromate, ascorbic acid and DPPH etc. used in the experiments are purchased from Merck and Fizmerk Pvt. Ltd. and used without additional purification**.**

5.1.2 Plants Selected

The green leaves different unique plants will be collected from the University Campus/surroundings at Hamirpur Himachal Pradesh.

5.2 Methodology

5.2.1 Preparation of Glassware Cleaning Agent

Dissolve around 5 gm of potassium dichromate and approximately 20 mL of sulfuric acid in 100 mL of water. Add concentrated sulfuric acid carefully while stirring continuously and introduce a small amount of nitric acid into the solution mixture. The resulting solution was utilized for the cleaning of glassware.

5.2.2 Preparation of leaf extract

The fresh leaves will be collected from the vicinity of Career Point University Hamirpur (H.P.). After thorough washing with tap water and subsequent rinsing with distilled water to eliminate dust, microorganisms, particles and other impurities. The cleansed leaf will be dried to eliminate surface moisture. The accurately weighed crushed leaves powder will be placed in a round-bottom flask and heated through rota mantle at 70°C. The resulting filtered plant extract solution will be employed in synthesizing NCs. This plant extract was stored in refrigerator for future use.

5.3 Synthesis of microwave assisted Metal Oxide

The synthesis will be done with microwave-assisted green method by using leaf extract as environmentally friendly reducing agent. For synthesis, different metal oxide will be mixed in varying ratios followed by the addition of leaf extracts and pH adjustment with

NaOH (1M). Microwave (2.45 GHz and 700 W) irradiation for 10 minutes will induce colour change, indicating NCs formation. Further it will be washed and dried (70-80 °C). Finally crushed to powdered form for further use. Similar synthesis will be achieved for different composition followed by washing and drying steps. This method offers an eco-friendly approach to nanocomposite synthesis, contributing for sustainable materials research.

5.4 Characterization of synthesized NCs

The green synthesized co-doped ZnO NPs will be characterized by XRD to determine their crystallite size, crystal structure, lattice parameters, etc. The band gap of synthesized NPs will be measured with UV-Vis spectroscopy and functional groups present inside the NPs will be identified with Fourier transform infrared spectroscopy (FTIR). The SEM, EDX and TEM analysis will be used to determine the **s**urface morphology and particle size.

5.5 Photocatalytic activity

To assess the photocatalytic activity of the synthesized NCs in degrading MO and MB, 100 mL aqueous solutions containing 10 ppm of each dye will be used (Xiong et al., 2017). Different amounts of synthesized nanocomposites were will be added to the solutions. The solutions will be stirred on a magnetic stirrer in the dark for few minutes to allow any adsorption on the synthesized catalyst. After this dark adsorption period, 5 mL of the solution will be withdrawn and labeled as the "0 min" sample. The remaining solution will be exposed to UV light illumination using a 125 W mercury vapor lamp, with photocatalyst. The degradation of the dyes will be monitored at regular intervals of few minutes until degraded for each amount of NCs. UV-visible spectrophotometry will be employed to track the decomposition of the dyes by measuring the changes in absorbance. After completing the reaction, the percentage degradation will be calculated using a specific equation (A et al., 2020; Balouiri et al., 2016).

$$\% \text{ Degradation} = \frac{(A_0 - A_t)}{A_0} \times 100$$

Where Ao represents the absorbance of the pure dye solution and A_t represents the absorbance of the reaction mixture at time t**.**

5.6 Antioxidant activity

The radical scavenging activity of different NCs will be assessed using the DPPH assay (Kumar et al., 2023). Various concentrations of the synthesized catalyst will be combined with a 0.1 mM DPPH solution in methanol followed with addition of NCs,, the violet color of the DPPH radical solution faded to colorless. After a specified incubation period, absorbance at 517 nm may be recorded, with ascorbic acid serving as the standard. A reduction in absorbance will reflect the enhancement in DPPH radical-scavenging activity. The efficacy of scavenging DPPH free radicals will be determined using the following equation.

$$\% \text{ inhibition} = \frac{(A_0 - A_1)}{A_0} \times 100$$

Absorbance of the solution denoted by A_1, while absorbance of the control solution denoted by A_0.

5.7 Antimicrobial activity

Antimicrobial activity of synthesized NCs will be tested by using well diffusion method against different positive and negative gram microbes or with some segment of fungus. Bacterial strains/fungus will be purchased from the entitled Institute of Microbial Technology (IMTECH).

6. Thesis Outline

The thesis will be comprised of following chapters:

Chapter 1 (Introduction): It will constitute of the brief account about nanotechnology along with characteristics of metal oxide NPs and NCs with plant extract. It will also include the objectives of proposed study.

Chapter 2 (Review of Literature): This chapter will deal with intense literature with context to the synthesis, characterization and applications of bimetallic oxide NCs. It will also include the research gap identification.

Chapter 3 (Materials and Methods): This chapter will comprise the detail of materials, microwave assisted green synthesis, characterization techniques and applications of transition metal oxide NCs.

Chapter 4 (Results and Discussion): This chapter will include the reports regarding physical and chemical properties of transition metal oxide NCs along with analysis and comparison of the findings of work.

Chapter 5 (Summary and Conclusion): It will include the overall summary and conclusion of the entire study with regard to work in context to the objectives cited.

Chapter 6 (References): Elsevier- Harvard style.

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CERTIFICATE
(SYNOPSIS APPROVAL CERTIFICATE)

This is to certify that the Synopsis/Revised Synopsis for Ph.D. Thesis titled, "**Green Synthesis, Characterization and Applications of Transition Metal Oxide Nanocomposites using Microwave Assisted Method**", submitted by Mr. Shakti Singh to the Career Point University, Hamirpur (H.P.), India in partial fulfillment of the requirements for the degree of DOCTOR OF PHILOSOPHY in the Department of Physics, School of Basic and Applied Sciences has been approved by the Research Advisory Committee (RAC) after an Oral Examination of the student on dated **15.05.2025**.

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